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# The Electron Microscopic and Micro-diffraction Studies of the Changes in Specimens

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CITATION:

Sasaki, Nobuji ...[et al]. The Electron Microscopic and Micro-diffraction Studies of the Changes in Specimens. Bulletin of the Institute for Chemical Research, Kyoto University 1954, 32(2): 87-90

ISSUE DATE:

1954-03-31

URL:

<http://hdl.handle.net/2433/75425>

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very slack in comparison with the case where the focal length of the projector lens is fixed. The experimental values of  $L_f$  prove well these relations, and the accordance between the two curves for  $L_f$  is very good as shown in Fig. 1.

The  $L_f$ - $k$  relation is also investigated under a condition of fixed exciting current  $i$  and varying the beam potential (V). The change of  $L_f$  against the beam potential  $V$  as shown in Fig. 2. The accordance between the calculated and observed values is pretty well, and it is obvious from the results that the camera length  $L_f$  has a slight increasing tendency as the beam potential increases.

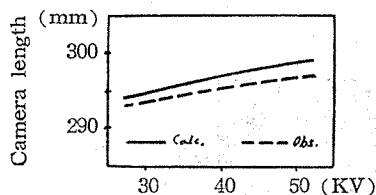


Fig. 2.  $L_f$ - $V$  relation.

In both cases, the stability of the camera length for the electron diffraction is rather well, when a three-stage electron microscope is used as an electron micro-diffraction camera.

The rotation angle ( $\varphi$ ) in the direction of orientation caused by the intermediate lens, between the electron image and its diffraction pattern is also investigated, and it satisfies the following relation

$$\varphi = \pi k' i n / \sqrt{V} \quad (3)$$

where  $k'$  is rather 0.6 than 0.8 as a weak lens with a pole piece of large diameter and gap separation is used for intermediate lens. Practically, it is suitable for estimating the rotation angle to be  $1^\circ$  as the exciting current  $i$  of the intermediate lens varies by 1 mA, when the number of coil turning  $n$  is about 21,000.

## 10. The Electron Microscopic and Micro-diffraction Studies of the Changes in Specimens

Nobuji SASAKI and Ryuzo UEDA

(Sasaki Laboratory)

A continual observation of the progress of change of a solid substance undergoing during a physical or chemical treatment by means of the electron microscopy accompanied by the electron diffraction method would provide valuable informations for the clarification of the nature of the process.

The specimen in the electronmicroscope is inevitably more or less affected by its own electron beams for observation. Many reports have been published on these effects upon the shape, the chemical composition, and the crystal structure. But only a very few are concerned with the integrated observation that we envisage.

To effect a change deliberately by some other means, either chemical or physical, an additional device is necessary. We have reported on the specimen-treating adaptor for the electronmicroscope, by which we can first observe a freshly prepared specimen without exposing it to injuring action of air and secondly take micrographs at one and the same part of a specimen before and after it is subjected to a certain physical or chemical treatment in order to detect by comparing them even minute changes the

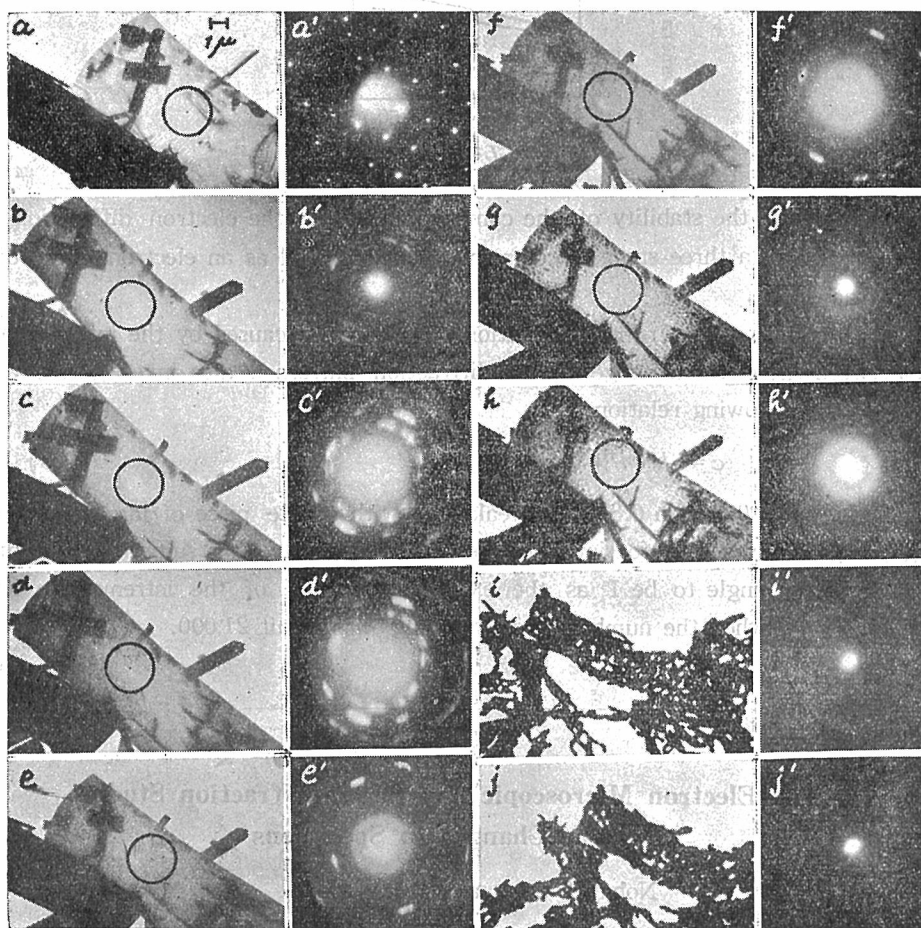


Fig. 1. Progress of reduction in a  $\text{McO}_3$  single crystal. The original crystal (a) was reduced by hydrogen successively: (b) for 5 min at  $500^\circ\text{C}$ ; (c) for 10 min. at  $500^\circ\text{C}$ ; (d) for 5 min. at  $600^\circ\text{C}$ ; (e) for 10 min. at  $650^\circ\text{C}$ ; (f) for 30 min. at  $650^\circ\text{C}$ ; (g) for 5 min. at  $700^\circ\text{C}$ ; (h) for 15 min. at  $700^\circ\text{C}$ ; (i) for 20 min. at  $700^\circ\text{C}$  and (j) for 10 min. at  $700^\circ\text{C}$ .

Each diffraction pattern belongs to the part indicated by a circle in the corresponding micrograph.

treatment has effected. (*Rev. Sci. Instr.* 23 136 (1952))

With this adaptor attached to a three stage electronmicroscope, micrographs and micro-diffraction patterns of a certain chosen point of a  $\text{MoO}_3$  single crystal were taken successively in the course of reduction by hydrogen (Figs. 1 and 2).

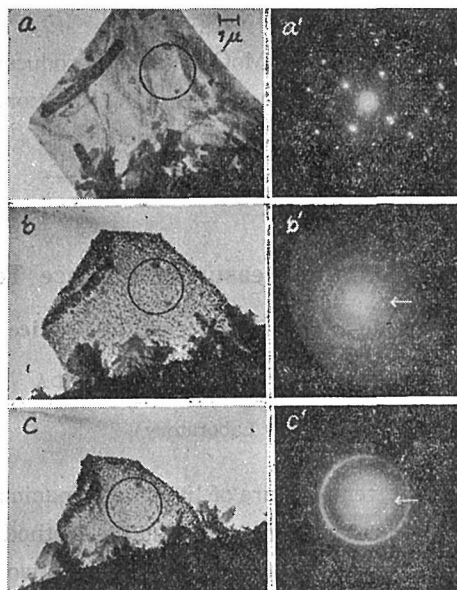


Fig. 2. Progress of reduction in a  $\text{MoO}_3$  single crystal. The original crystal (a) was reduced by hydrogen (b) for 10 min. at  $500^\circ\text{C}$ , then (c) for 10 min. at  $600^\circ\text{C}$ .

1) Since the chemical substances produced successively by the reduction in a definite small region of the crystal can be identified by micro-diffraction patterns, the micrographical change at that point can be correlated with the progress of the chemical change.

2) When more than two compounds are produced simultaneously by the chemical reaction, the correlation between the micrograph and the micro-diffraction pattern can be achieved by performing many-sided experiments and taking a possible reaction mechanism into consideration.

3) The micro-diffraction ring corresponding to the (110)-plane of  $\text{MoO}_2$  changes into diffused one in the earliest stage of reduction, which indicates that the (110)-plane is the most easily affected by hydrogen. (see Fig. 2-note the ring marked with an arrow).

4) a) In the course of reduction we often observed that, while the original single crystal showed no sign of change in the electronmicrograph, the diffraction pattern revealed its complete transformation into an aggregate of minute crystals (Fig. 1 a, a', b and b').

b) In a certain stage of reduction a remarkable change in diffraction pattern can

often occur without being accompanied by a corresponding morphological change. The change in diffraction pattern is usually observed earlier than the change in electronmicroscopic pattern. This means that the atomic rearrangement has occurred without affecting the external shape and the specimen at first assumes a "Pseudo-structure" or the external change is beyond the resolving power of the electron-microscope.

5) Diffraction patterns of Mo and MoO<sub>2</sub> particle produced by reduction within minute single crystal of MoO<sub>3</sub> show some preferred orientation. Such an information can only be obtained by the micro-diffraction method.

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## 11. A New Method for Measuring Surface Temperature

### A Wide-range Self-recording Device

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The knowledge of surface temperature of living and inanimate objects has become increasingly important in science and industry. But the method of measurement seems not to have been well standardized (reference is made to two articles in the book, "Temperature, Its Measurement and Control in Science and Industry", American Institute of Physics, Reinhold Publishing Corporation, New York, 1941, 1) "Measurement of Surface Temperatures", F.C.Houghten and H. T. Olson, p. 855, 2) "Temperature of Incandescent Lamps", W. E. Forsythe and E. M. Watson, p. 1188).

The method presented here makes use of a thermocouple (in principle a resistance thermometer or even a liquid-in-glass thermometer can be used as well) whose measuring junction can be adjusted to any temperature. The temperature of the junction indicated by a millivoltmeter in the couple circuit is also the temperature of the surface, if the junction temperature has been so adjusted that the needle of the meter shows no deflection, when the junction makes a light brief contact with the surface (for a wet or liquid surface a non-wetting junction should be used).

Surface temperatures measured by this method can be recorded and the principle of the method thereby clearly visualized.

The alternating current for heating the measuring junction of a thermocouple is periodically and smoothly varied between two fixed values by moving slowly the iron core A to-and-fro in an inductor B in the circuit (Fig. 1). The temperature of the junction C oscillates between two temperatures  $T_1$  and  $T_2$ . A galvanometer (period 0.2 sec, internal resistance 37.3 ohms, and the sensitivity  $5 \times 10^{-8}$  amp/mm.) D in the thermocouple circuit reflects a light spot from source S into a photographic film moving vertically. The record is a sort of sine curve which oscillates between